Designation: B 657 – 92 (Reapproved 2000)

Standard Test Method for Metallographic Determination of Microstructure in Cemented Tungsten Carbides[1]

This standard is issued under the fixed designation B 657; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers apparatus and procedures for the metallographic determination of microstructures in cemented tungsten carbides.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Precautions applying to use of hazardous laboratory chemicals should be observed for chemicals specified in Table 1.

2. Referenced Documents

2.1 *ASTM Standards:*

B 665 Practice for Metallographic Sample Preparation of Cemented Tungsten Carbides[2]

2.2 *ISO Standard:*

ISO 4499 Hardmetals—Metallographic Determination of Microstructure[3]

3. Terminology

3.1 Definitions of Symbols:

Symbol	Definition
α phase	tungsten carbide
β phase	binder (for example, Co, Ni, Fe)
γ phase	carbide of a cubic lattice (for example, TiC, TaC, NbC) that may contain other carbides (for example, WC) in solid solution
η-type phases	multiple carbides of tungsten and at least one metal of the binder

4. Significance and Use

4.1 The microstructure of cemented tungsten carbide affects the material's mechanical and physical properties. This is not intended to be used as a specification for carbide grades, producers and users may use the microstructural information as a guide in developing their own specifications.

5. Apparatus

5.1 *Metallographic Microscope* capable of magnifications up to 1500 times.

5.2 Ordinary metallurgical laboratory equipment.

5.3 Equipment for specimen preparation as outlined in Practice B 665.

6. Specimen Preparation

6.1 A suitable procedure is described in Practice B 665.

7. Procedure

7.1 Examine the microstructure by gradual development of the phases by etching. Examples of suitable etching techniques are given in Table 1.

7.2 Determine the presence of η-type phases by lightly etching the section with Technique 1 (see Table 1). Phases of η-type are colored orange to brown. γ-Phase may etch lightly, while the other phases remain unetched. Etching by Technique 1 does not preclude subsequent etching by Techniques 2 or 3. Examine the entire section at low magnification and, if necessary, at magnifications up to 1500 times. Note and record the existence of η-type phases and their distribution.

7.3 Determine the presence of γ phases by etching with Technique 2 (Table 1). This phase appears light yellowish brown and has a typically rounded shape (see Fig. 1). Examine the etched section and note and record the existence of a γ phase. Estimate and record its size according to Fig. 1 as γ–fine, γ– medium, or γ–coarse.

7.4 Determine the presence of α phase by etching the section with Technique 3, or in case γ phase is present, with Technique 2. The α phase appears gray. Examine the etched section and note and record the presence of α phase. Estimate and record its size according to Fig. 2 as α–fine, α–medium, or α–coarse.

7.5 Identify the β phase after etching the surface by Technique 3 (Table 1). This phase remains white.

NOTE 1—This procedure follows essentially ISO 4499.

[1] This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.06 on Cemented Carbides.

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

8. Report

8.1 The report shall include complete identification of the specimen and the results obtained.

9. Precision and Bias

9.1 The nature of this test method precludes any statement of precision or bias.

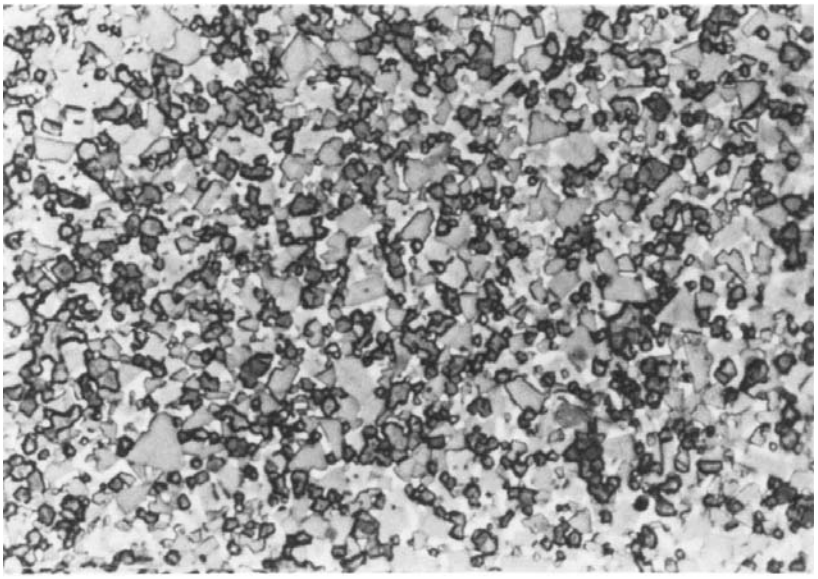
10. Keywords

10.1 cemented carbides; hardmetals; microstructure; powder metallurgy

TABLE 1 Etching Techniques

NOTE 1—The separate solutions of potassium hexacyanoferrate (III) and potassium or sodium hydroxide may be stored for a long time, but must be freshly mixed each day when used.

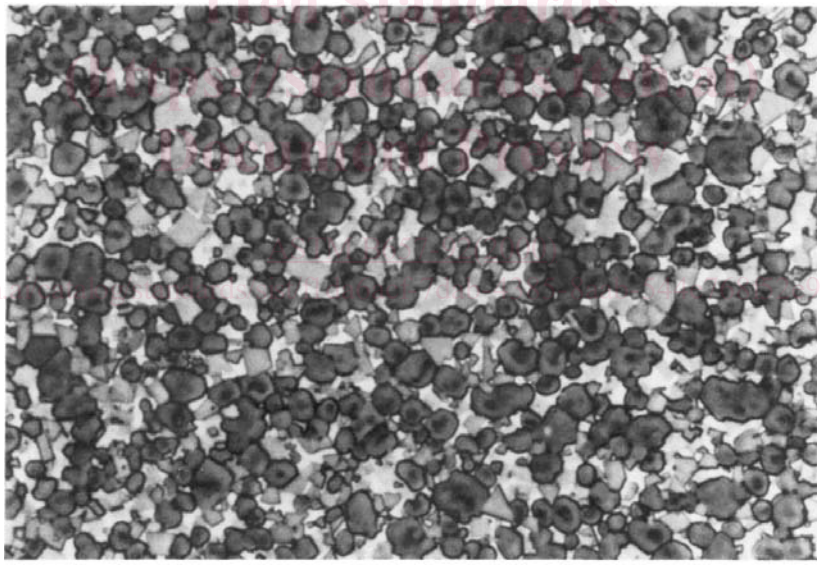
Etching Technique	Composition of Etchants	Conditions of Etching	Objective of Etching
1	A Freshly prepared mixture of equal quantities of 10 % (mass/mass) aqueous solutions of $K_3Fe(CN)_6$ (III) (potassium ferricyanide) and potassium or sodium hydroxide	Etch in mixture A at approximately 20°C for 2 to 10 s. Flush the test-piece section with water immediately, without removing the oxide layer. Dry the surface carefully with acetone or alcohol without wiping.	Identification of η phases
2	A Same as 1A B A mixture of equal volumes of concentrated hydrochloric acid and water	Etch at approximately 20°C in mixture A for 3 to 4 min. Then wash in water and etch in mixture B for approximately 10 s. Next wash in water, then in alcohol and dry the test-piece section. Finally, etch in mixture A for approximately 20 s.	Identification of γ phase
3	A Same as 1A	Etch in mixture A at approximately 20°C for 3 to 6 min.	Identification of α phase



γ -fine

(This figure is reprinted from ISO 4499.)

FIG. 1 γ Phase 1500×.



γ -medium